

# PATENT SPECIFICATION

(11) 1 579 934

1 579 934

- (21) Application No. 42686/76 (22) Filed 14 Oct. 1976  
 (23) Complete Specification filed 3 June 1977  
 (44) Complete Specification published 26 Nov. 1980  
 (51) INT CL<sup>3</sup> A61K 7/48  
 (52) Index at acceptance  
     C3V AP  
     A5B 161 FH  
     C3W 100 215C 217 225C  
     C3Y B101 B230



- (72) Inventors **RAYMOND GUY HARRISON** and  
**PATRICIA MARY HAGARTY**

## (54) SKIN-CARE COSMETIC PRODUCTS

(71) We, **BEECHAM GROUP LIMITED**, a British Company of Beecham House, Great West Road, Brentford, Middlesex, do hereby declare the invention for which we pray that a patent may be granted to us and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to a skin-care product of cream-like or lotion consistency, having a high water content and containing tiny polymer particles. The product may be used as a moisturising cream or lotion and has the advantage of leaving the skin with a smooth feel.

Moisturising creams and lotions are widely used as skin cosmetics. They are generally unpigmented perfumed oil-in-water emulsions (or, less commonly, water-in-oil emulsions) with a relatively high water content of from about 60—90% by weight. Such creams are absorbed by the upper layers of skin cells, swelling them to some extent, but otherwise leaving essentially no residual effect on the skin other than a slight perfume. They differ from so-called vanishing creams in that they contain essentially no free fatty acid.

Powder products have traditionally been used for skin smoothing purposes, the powder filling pores and fine wrinkles on the skin surface. With the advent of polymer technology, proposals were made to substitute polymer particles for the powders, such as talc, used in these products. Specifications 774029, 818425, 1059970, 1093108, 1141994 and 1202796 all refer to the possible use of polymer particles in cosmetics.

During the course of our investigations into the use of polymer particles in moisturising creams or lotions we found that spherical or substantially spherical particles in the range 5 to 15 microns could be incorporated in amounts from about 3% to about 10% by weight of the cream or lotion without adversely affecting the stability and other desirable properties. Moreover, despite the very small amount of polymer present, the resulting product rendered the skin particularly smooth and silky.

Accordingly, the present invention provides a skin-care product comprising an unpigmented oil-in-water (or water-in-oil) emulsion containing from 60—90% by weight of water and having dispersed therein from 3 to 10% by weight of substantially spherical synthetic polymer or copolymer particles having an average particle size in the range 5 to 15 microns, the oil being a non-volatile oil, as herein defined.

As used herein, the term "non volatile oil" means an oil (including viscous or semi-solid liquids) which, when applied as a thin film on a surface maintained at 37°C, does not evaporate to an extent greater than 10% by weight of the applied quantity in a period of 3 hours. Examples include mineral oil and petroleum jelly.

Preferably the product should contain a perfume, and preferably there should be substantially no free fatty acid present.

Usually the product will contain from 3 to 5% by weight of the polymer or copolymer particles.

Preferably the polymer or copolymer particles shall have an average size of from 5 to 7 microns.

Preferably, substantially none of the polymer or copolymer particles should exceed about 50 microns, and best results are achieved when there are substantially no particles of a size greater than 15 microns.

The polymer or copolymer particles should preferably not swell to any great

extent in water or the oil used in the product. Within this limitation cross-linked polystyrene or polymethyl methacrylate microspheres would be suitable. However, the preferred polymer is polyethylene.

The following are Examples of the formulae of certain moisturising creams and lotions according to the invention:—

#### Moisturising Lotion (oil-in-water)

Formula	% w/w	% w/w
Mineral Oil	20.00	20.00
Stearic Acid	4.00	4.00
Triethanolamine	1.30	1.30
Preservatives	qs	qs
Fragrance	qs	qs
Distilled water	71.70	65.70
*Polymist B-6	3.00	9.00
	100.00	100.00

#### Moisturising Cream (water-in-oil)

Formula	% w/w	% w/w
Mineral Oil	22.00	22.00
Beeswax	5.00	5.00
Petroleum jelly	10.00	10.00
Cetyl alcohol	2.00	2.00
Borax	0.50	0.50
Magnesium Sulphate	0.20	0.20
Preservatives	qs	qs
Fragrance	qs	qs
*Polymist B-12	3.00	9.00
Distilled water	57.30	51.30
	100.00	100.00

\*Polymist is a Trade Mark of Allied Chemicals; Station House, Stamford New Road, Altrincham, Cheshire WA16 1EP, England.

Polymist B-6 and B-12 are high density (0.96 gm/cc by ASTM D-1505) polyethylene microspheres having an average particle size of 6 $\mu$  and 12 $\mu$  respectively, a particle size range of from 2—12 $\mu$  and 2—24 $\mu$  respectively with less than 2% and about 50% respectively of the particles larger than 10 $\mu$ .

#### WHAT WE CLAIM IS:—

1. A skin-care product comprising an unpigmented oil-in-water (or water-in-oil) emulsion containing from 60—90% by weight of water and having dispersed therein from 3 to 10% by weight of substantially spherical synthetic polymer or copolymer particles having an average particle size in the range 5 to 15 microns, the oil being a non-volatile oil as herein defined.

2. A skin care product as claimed in claim 1 including a perfume and containing substantially no free fatty acid.

3. A skin care product as claimed in claim 1 or claim 2 containing from 3 to 5% by weight of the polymer or copolymer particles.

4. A skin care product as claimed in any one of the preceding claims wherein substantially none of the polymer or copolymer particles have a particle size greater than 15 microns.

5. A skin care product as claimed in any one of the preceding claims wherein the polymer or copolymer particles are polyethylene, or cross-linked polystyrene or polymethylmethacrylate microspheres.

Agent for the Applicants,  
H. B. DAWSON.

Printed for Her Majesty's Stationery Office, by the Courier Press, Leamington Spa, 1980  
Published by The Patent Office, 25 Southampton Buildings, London, WC2A 1AY, from which copies may be obtained.

# PATENT SPECIFICATION

(11) 1370699

- (21) Application No. 60146/71 (22) Filed 23 Dec. 1971  
 (31) Convention Application No. 2063896 (32) Filed 28 Dec. 1970 in  
 (33) Germany (DT)  
 (44) Complete Specification published 16 Oct. 1974  
 (51) International Classification A61K 7/00 9/06  
 (52) Index at acceptance  
 A5B 26Y 344 34Y 351 35Y 401 40Y 771  
 (72) Inventors RUDOLPH TUMA and FRITZ NEUWALD



## (54) IMPROVEMENTS IN OINTMENT BASES

- (71) We, DYNAMIT NOBEL AKTIENGESellschaft, a joint stock company organised under the laws of Germany, of Postfach 1209, 521 Troisdorf, Germany, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—
- It is known that coconut oil containing longer-chained fatty acid and lauric acid is not suitable for use as an ointment base due to the strong dependence of the consistency on the temperature.
- In German Patent 1,090,824, it is proposed to mix 50 to 75 parts by weight of a mixture of fatty acids with 8 to 12 carbon atoms in the molecule with 50 to 25 parts of a fatty acid with 18 to 22 carbon atoms in the molecule and esterify the mixture obtained with glycerine. In this way an ointment base is obtained which, like soft paraffin, has a very good spreadability, but whose consistency at temperature above 35°C is no longer satisfactory.
- According to U.S. Patent 2,628,187 liquid paraffin is brought into an ointment-like consistency by working in polyethylene. However the use of hydrocarbons in pharmaceuticals and cosmetics is limited due to the hydrophobic properties of these products. Attempts have also been made to give liquid triglycerides e.g. almond oil, an ointment-like consistency by means of additives and to this end natural waxes such as spermaceti and beeswax have been proposed. Such mixtures of natural waxes or tallow with vegetable oil do not, however, fulfil the demands made on ointment bases due to their lack of stability.
- It has hitherto proved impossible to convert fatty acid esters with a liquid consistency containing no unsaturated fatty acids into a spreadable ointment base.
- We have now developed an ointment base which remains spreadable over a wide temperature range of at least 0—40°C and which is not prone to become rancid during storage, based on glycerine esters of saturated fatty acids with 8 to 12 carbon atoms in the molecule.
- One aspect of the present invention provides an ointment base comprising an intimate mixture a saturated triglyceride wherein the fatty acid residues each contain 8—12 carbon atoms and 5—20% by weight of a gelling agent effective to give said triglyceride the consistency of a gel over at least the temperature range from 0° to 40°C, said gelling agent being polyethylene having an average molecular weight of 1,000 to 20,000 or an ethylene/vinyl acetate copolymer.
- A suitable copolymer comprises e.g. 30—70 parts by weight ethylene and 70—30 parts complementary vinyl acetate, and preferably has a molecular weight in the range 1,000 to 10,000. An addition of waxes e.g. a microcrystalline wax (a synthetic wax of branched chain C<sub>10</sub>—C<sub>26</sub> hydrocarbons) or beeswax up to 20% by weight causes a further increase in the stability.
- It is preferred that the ointment base has a viscosity of 180—500 poise at 20°C. It will be understood that no significance chemical reaction takes place between the triglyceride and the gelling agent.
- The ointment base produced has an excellent compatibility with the skin because the main component thereof is a fatty substance. It readily penetrates the skin so that medicaments incorporated in the base are well absorbed. In addition the ointment base has a very good stability not possessed by natural fats or oils due to their content of unsaturated fatty acids.
- According to another aspect of the invention the incorporation of the gelling agent, such as polyethylene with an average molecular weight from 1,000 to 20,000 may be performed by dissolving it in the triglyceride at an elevated temperature, e.g. while briefly heating the mixture to 60—90°C accompanied by stirring, preferably followed by heating under vacuum at 100—140°C. When solution occurs, the mixture is cooled, e.g. to about 50°C by passage over an appropriate cooling roller, whereby a gel is formed and the mass

solidifies. The solidified mass is milled on a roller mill and a base with an ointment-like consistency is obtained.

5 As triglyceride starting material we prefer to use materials having the following characteristics: acid number less than 1; saponification number 220—345; iodine number less than 1.

10 The rheological properties as well as the temperature stability of the ointment bases according to the invention can be further improved by adding waxes advantageously in a proportion of up to 20% by weight e.g. 5—20% by weight. One may use e.g. 3 to 15 10% by weight of beeswax or spermaceti or 5 to 20% by weight of microcrystalline waxes. Alternatively or in addition one may add up to 2%, e.g. 1—2% by weight of a metallic soap having gelling properties, especially 20 aluminium monobasic salts of fatty acids having 14—18 carbon atoms, e.g. aluminium monostearate. The liquid triglyceride does not separate from the ointment base even after prolonged storage and the consistency of the 25 base remains virtually unchanged in the temperature range from 0 to 40°C.

30 The ointment bases according to the invention are suitable as vehicles for the most varied pharmaceutical agents because a homogeneous distribution of the active ingredients can be obtained without special mixing procedures and generally demixing does not occur even after prolonged storage.

35 The ointment bases according to the invention can also be successfully used in cosmetics where they simultaneously protect the skin from drying as a result of their lubricating action, without however leaving an unwanted grease film on the skin.

40 In order that the invention may be better understood, the following Examples are given by way of illustration only. The viscosity measurements were made by the Fryklöf method, using a Brookfield viscometer.

#### 45 Example 1

60 68.5 g of a triglyceride mixture of  $C_8$ — $C_{12}$  fatty acids (viz. 45% caprylic acid, 8% lauric acid and 47% caproic acid, the triglyceride having the following characteristics: acid number 0.08; saponification number 332; hydroxyl number 10; iodine number 0.5) is heated to 80°C in a three-necked flask accompanied by stirring and 1.5 g of aluminium monostearate is stirred in. After 55 placing the mixture under a reduced pressure of 20 Torr it is slowly heated to 100°C. After formation of a gel the vacuum is released by addition of nitrogen and 10 g of polyethylene wax (molecular weight 12,000) 60 as well as 20 g of a microcrystalline wax are added. Subsequently the pressure is again reduced to 20 Torr and the temperature is adjusted to 130°C over 1/2 hour. As soon as this temperature is reached, intensive stirring

takes place and the reaction mixture is cooled to 110°C and subsequently, by means of a cooling roller, to a temperature below 50°C. After milling on a roller mill, an ointment base similar to petroleum jelly is obtained with the following characteristics: acid number 1.8; saponification number 231; hydroxyl number 13; iodine number 0.5.

The viscosity of this ointment base is about 210 p (20°C).

#### Example 2

80 g of the triglyceride mixture of Example 1 are stirred and processed as in Example 1 with 1.5 g of aluminium monostearate, 10 g of polyethylene wax (average molecular weight 15,000) and 10 g of bleached wax under the same conditions. The resulting ointment base has the following characteristics: acid number 2.4; saponification number 238; hydroxyl number 11; iodine number 1.7.

The viscosity is about 345 p (20°C).

#### Example 3

85 g of triglyceride mixture of  $C_8$ — $C_{11}$  fatty acids (viz. 41% caprylic acid, 14% lauric acid and 45% caproic acid, the triglyceride having the following characteristics: acid number 0.05; saponification number 347; hydroxy number 5; iodine number 0.3) is stirred at 85°C. and processed as in Example 1 with 15 g of polyethylene wax (average molecular weight 10,000) under the same conditions.

The resulting ointment base has the following characteristics: acid number 1.2; saponification number 255; iodine number 1.2. The viscosity of this ointment base is about 320 p (20°C).

#### Example 4

85 g of the triglyceride mixture of Example 1 is processed as in Example 2 with 5 g of a copolymer of ethylene and vinyl acetate (average molecular weight 18000 and containing 55 weight % vinyl acetate) and 10 g of a microcrystalline wax; 1.3 g of aluminium monopalmitate was used in place of the aluminium monostearate of Example 2 and stirring under vacuum was performed at 115°C.

The resulting ointment base has the following characteristics: acid number 2.9; saponification number 246; hydroxyl number 25; iodine number 1; viscosity 290 p (20°C).

#### Example 5

160 g of a triglyceride mixture (the fatty acid content being 38% caprylic acid, 17% lauric acid and 45% caproic acid and the triglyceride having the following characteristics: acid number 0.05; saponification number 344; hydroxyl number 8; iodine number 1) are processed with 20 g of polyethylene wax, molecular weight 15,000, accompanied by

65

70

75

80

85

90

95

100

105

110

115

120

125

stirring and heating under the conditions of Example 1. No aluminium salt was present. After reaching the solution temperature of 130°C within 20 minutes, with the aid of a cooling roller the mixture is brought to a temperature below 50°C and subsequently, after processing on a roll mill petroleum jelly-like ointment base with the following characteristics is obtained:

10	Acid number	2.5
	saponification number	242
	Iodine number	1.5
	Viscosity	360 p

#### WHAT WE CLAIM IS:—

- 15 1. An ointment base comprising in intimate mixture a saturated triglyceride wherein the fatty acid residues each contain 8—12 carbon atoms and 5—20% by weight thereof of a gelling agent effective to give said triglyceride the consistency of a gel over at least the temperature range from 0° to 40°C, said gelling agent being polyethylene having an average molecular weight from 1,000 to 20,000 or an ethylene/vinyl acetate copolymer.
- 25 2. An ointment base according to claim 1 wherein said copolymer comprises 30—70 parts by weight ethylene and 70—30 parts by weight complementally vinyl acetate and has a molecular weight in the range 1,000 to 10,000.
- 30 3. An ointment base according to claim 1 or 2 including up to 20% by weight of a wax.
- 35 4. An ointment base according to claim 3 including 3—10% of spermacetic or bees-wax.
5. An ointment base according to claim 3 including 5—20% of a microcrystalline wax.
- 40 6. An ointment base according to any of the preceding claims including up to 2%

by weight of a metallic soap having gelling properties.

7. An ointment base according to claim 6 wherein said metallic soap is a monobasic aluminium salt of a fatty acid having 14—18 carbon atoms.

8. An ointment base according to claim 6 wherein said metallic soap is aluminium monostearate.

9. An ointment base according to claim 1, substantially as hereinbefore described and as illustrated with reference to any of Examples 1—4.

10. An ointment base according to claim 1, substantially as hereinbefore described and as illustrated with reference to Example 5.

11. A method for the preparation of the ointment base defined in claim 1 which comprises dissolving said gelling agent in said triglyceride at an elevated temperature and cooling the mixture to form a gel.

12. A method according to claim 11 wherein the mixture is heated at 60—90°C and subsequently heated under vacuum at 100—140°C.

13. A method according to claims 11 or 12 wherein the product is subsequently milled on a roller mill.

14. An ointment base made by the method of any of claims 11—13.

15. A cosmetic preparation comprising an ointment base according to any of claims 1—10 or 14.

16. A pharmaceutical preparation comprising as vehicle an ointment base according to any of claims 1—10 or 14.

For the Applicants,  
FRANK B. DEHN & CO.,  
Chartered Patent Agents,  
Imperial House,  
15/19 Kingsway,  
London WC2B 6UZ.